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Bi-Monthly Progress Report

P-A2476-6

PREPARATION AND EVALUATION
OF HIGH PURITY BERYLLIUM

by

G. E. Spangler
M. Herman
V. V. Damiano
E. J. Andt
D. B. Hoover

September 2 to November 1, 1961

Prepared for

DEPARTMENT OF THE NAVY
Bureau of Naval Weapons

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Contract No. N0w 61-0221-a

THE FRANKLIN INSTITUTE
LABORATORIES FOR RESEARCH AND DEVELOPMENT
PHILADELPHIA PENNSYLVANIA

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ABSTRACT OF CURRENT RESEARCH

The zone refining of one inch diameter Brush vacuum cast beryllium bar was continued, along with the zone refining of smaller diameter vacuum distilled and Pechiney SR grade bars.

Three single crystal specimens of varying purity content were tested in tension. Vacuum distilled material which was subsequently zone melted seven passes exhibited a value of 400 psi for the critical resolved shear stress on the basal plane.

Additional zone melted polycrystalline was prepared by hot rolling and recrystallizing four pass single crystal beryllium. Two different orientations with respect to the rolling plane were used. Tensile tests and bend tests were performed, but the results to date are inconclusive.

Preliminary examinations have been made of zone refined beryllium by electron transmission microscopy. It has been observed that the dislocations do not move under the influence of the electron beam alone as they have been observed to move in other metals. When the specimen was heated to 200°C, however, and cycled over a narrow range of temperatures, a slow movement of the dislocations was observed.

SCOPE OF RESEARCH

To prepare high purity beryllium and to study its deformation and fracture characteristics.

ZONE REFINING

During the last two month period the zone melting of a one inch diameter Brush vacuum cast beryllium bar was continued. A total of ten passes have been made through this particular bar. Because several of the initial passes were made at rates greater than one half an inch per hour, one additional pass at one quarter inch per hour will be made through this bar to attempt to improve the purity attained. This material, in the form of a one inch diameter single crystal roughly six inches in length, will then be ready for evaluation. The emphasis of this evaluation will be on the properties of polycrystalline material, attained by mechanically deforming and recrystallizing the single crystalline material.

In the zone melting of smaller diameter (one quarter inch) bars, additional passes have been made both through several SR grade Pechiney bars as well as through the Nuclear Metals Inc. vacuum distilled bar. A total of five passes have been made on the initial SR grade bar and seven passes on the vacuum distilled material. Two additional SR grade bars have been subjected to two passes. These latter two bars are being zone refined in preparation for re-alloying the beryllium with selected impurity elements.

MECHANICAL TESTING

The critical resolved shear stress for basal glide was determined for three single crystals of varying levels of purity. The results of these tests, shown in Table I, again demonstrate a strong relation

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between the expected purity level, as estimated from starting purity and zone melting history, and the CRSS for basal glide. It is of interest to note that the value of 400 psi for the vacuum distilled, zone melted bar is the lowest value attained to date. It should also be noted that the value for the SR grade material is the lowest observed for what is essentially a starting material. The first two fast passes very likely did little more than permit evolution of the residual chloride which is normally removed by a vacuum melting operation.

TABLE I

Starting Stock	Zone Melting History	Orientation	CRSS on Basal Plane (psi)
NMI	1 pass- $1\frac{1}{2}$ " /hr.	$\chi_o = 50^\circ$	400
Vacuum Distilled	6 passes- $\frac{1}{2}$ " /hr.	$\lambda_o = 56^\circ$	
Pechiney SR Grade	2 passes-2" /hr.	$\chi_o = 48^\circ$	803
	1 pass- $\frac{1}{2}$ " /hr.	$\lambda_o = 52^\circ$	
Brush	2 passes- $1\frac{1}{2}$ " /hr.	$\chi_o = 74^\circ$	1070
Vacuum Cast	1 pass- $\frac{1}{2}$ " and $1\frac{1}{2}$ " /hr.	$\lambda_o = 74^\circ$	
1" Diameter	1 pass- $\frac{1}{2}$ " /hr.		

POLYCRYSTALLINE ZONE MELTED MATERIAL

An attempt was made to evaluate the tensile properties of polycrystalline material prepared from the initial four pass one inch diameter Brush bar. Additional bend tests were also performed. The polycrystalline material was prepared by hot rolling the jacketed one inch diameter single crystal material at 700°C, and then recrystallizing the worked material at a suitable temperature. Two sets of samples were prepared. For one set the rolling procedure was that described previously⁽¹⁾. For the second set the crystal was oriented with the basal plane

perpendicular to the rolling plane and parallel to the rolling direction. The rolling was all done in one direction with 5% reductions per pass up to a two to one reduction from the starting thickness followed by 10% reductions per pass to the final ten to one total reduction. The first set was recrystallized at 800°C for one half an hour and the second set at 750°C for two hours. In both cases the final mean grain diameter was roughly 70 microns.

It was observed that the rolling technique used for the first set resulted in a highly oriented structure. Examination with polarized light showed complete extinction of all grains for a full 360° rotation of the specimen. It was for this reason that a different rolling procedure was used for the second set, hoping to produce a less highly oriented structure. Examination of this material, however, showed extinction over much of the sample. Also, the grains which did not show extinction were in somewhat localized banded areas. It appeared that some recrystallization occurred during rolling such that the banded areas represented certain grains which were further deformed and then recrystallized to produce localized areas of different orientation.

For both sets the rolling resulted in sheets approximately one sixteenth of an inch thick. Blanks for the tensile specimens and bend specimens were cut from this sheet material using a wet abrasive cut off wheel. Gage sections were machined in the tensile specimens using the spark discharge machining technique. Both the bend specimens and the tensile specimens were then etched to remove about 0.010 inches from all surfaces.

In the bend tests, the more highly oriented sheet produced by the first rolling technique underwent a 15° bend about a one eighth inch mandrel. The sheet from the second rolling technique underwent a 30° bend, producing about 5% strain at the outer surface. The specimens each had a ten to one width to thickness ratio.

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It would appear that the reduction in the degree of orientation afforded by the second rolling technique did produce a beneficial effect upon the bending characteristics. The results of these tests, however, were inferior to those observed previously⁽¹⁾ so that it is difficult to conclude that these results were definitive.

The tensile tests were also inconclusive in that fracture occurred in the grips in each case. In order to prevent this the tensile specimen design is being modified to reduce the notch effect responsible for the observed fractures.

It is well understood that to attain the optimum polycrystalline properties in beryllium one must achieve a fine grained structure and, if possible, some degree of randomness in the crystal orientation from grain to grain. For these reasons plans are being made to investigate additional techniques for working down the one inch diameter single crystals. Preparations are also underway for determining more quantitatively the degree of preferred orientation by means of standard x-ray techniques in order to evaluate these various deformation techniques.

ELECTRON TRANSMISSION MICROSCOPY STUDIES

The examination of thin beryllium foils with the electron microscope is expected to yield considerable information regarding the behavior of dislocations under stress and ultimately lead to a better understanding of the flow and fracture characteristics of beryllium.

Many of the techniques for thinning metals developed in this laboratory⁽²⁾ will be used for the preparation of beryllium foils. This involves three stages: first the initial cutting of the specimen to the desired shape, size, and orientation; second the jet polishing of a depression in the surface in the desired location; and finally the electropolishing of the specimen to the point where thin areas are produced at the base of the depression.

In the first stage spark discharge techniques were used for the slicing and shaping operation. Thin sections ranging from 0.050 to 0.100" were cut from a one inch diameter, four pass zone refined beryllium crystal using cutting speeds of the order of 0.001"/min. Slices approximately .030" thick were then cut from the 0.100" sections producing specimens 0.030 x 0.100 x 3/4". These sections were then electropolished to a thickness of 0.020" and were then ready for the second stage.

Techniques are being developed for drilling and punching using spark discharge techniques. Small tensile specimens have been cut from 0.020" thick crystals. The specimens are rectangularly shaped and have the dimensions 0.020" x 0.100" x 0.500". A 0.040" hole is located at each end of the specimen for gripping.

In the second stage of the thinning operation the jet polisher recently assembled for the investigation of beryllium was used. The jet polisher is equipped with two calibrated mechanical stages for manipulating both the specimen and the jet. This allows the accurate placement of a depression at any desired location on the specimen within \pm 0.02 mm. A 20% HNO_3 solution is very effective as the electrolyte using currents of the order of 0.1 amp. for producing either holes or depressions.

In the third stage the depression is viewed through a microscope while the specimen is electropolished. A solution of 9 parts H_3PO_4 , 3 parts H_2SO_4 , 3 parts Ethanol, 3 parts glycerol is used as the electrolyte. The polishing operation is interrupted at the first appearance of a hole at the center of the depression. This is best accomplished by placing a well columnated beam of light behind the specimen and watching for the first breakthrough of light through the foil. Since the procedure of visually observing the specimen is generally long and tedious, a photoelectric cell was placed in the eyepiece of the microscope to sense the first breakthrough of light. At this time an alarm alerts the operator to remove the specimen from the electrolyte.

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Success in the use of the photoelectric technique depends upon minimizing the background reflection. It was found that an arrangement of the microscope in a horizontal position such that the specimen is viewed through a window in the side of a cell was best suited for this purpose. Areas adjacent to the hole have been found to be transparent to electrons, and dislocations and networks as seen in Figure 1 have been recorded.

The use of a special holder developed by Kear⁽³⁾ for the Phillips EM-100B electron microscope is anticipated. The holder is designed to pull specimens in tension and to heat specimens to 500°C while viewing the dislocations in the electron microscope.

Figure 1 shows the structure of a section of the one inch diameter four pass zone refined beryllium. The (0001) plane is approximately parallel to the surface of the film and dislocations and dislocation networks lying in the (0001) plane are seen. For convenience the $\langle 11\bar{2}0 \rangle$ slip directions have been designated AB, BC, and CA. Three-fold nodes typical of hexagonal nets are visible. These are believed to arise from the interaction of two sets of $1/3 \langle 11\bar{2}0 \rangle$ type Burgers vectors. The reaction $AB + BC \approx AC$ results in the formation of a third dislocation at the point of intersection.

Preliminary experiments have shown that the dislocations do not move under the influence of the electron beam alone as they have been observed to move in other metals. However when the specimen was heated to 200°C and cycled over a narrow range of temperatures slow movement of the dislocations was observed.



**Figure 1 - Electron Transmission Micrograph of Beryllium
Single Crystal (0001) approximately parallel
to plane of view**

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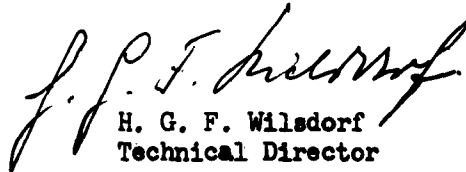
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Further experiments are anticipated along these lines now that the techniques for producing thin sections of beryllium have been successful.



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Approved by:



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